



*Institute of Paper Science and Technology
Atlanta, Georgia*

IPST Technical Paper Series Number 759

Measurement of Delignification Diversity Within Kraft Pulping Processes

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November 1998

Submitted to
AIChE Meeting
1998 TAPPI Pulping Conference
Montreal, Quebec, Canada
October 25–29

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MEASUREMENT OF DELIGNIFICATION DIVERSITY WITHIN KRAFT PULPING PROCESSES

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ABSTRACT

Measurements of the variation in kraft pulp lignin content have been the subject of intense interest for several years. Historically, macroscale variation has been determined in mill studies using hanging-baskets within batch digesters; at which time, lignin content variability was observed and related to chip location and thickness. On a smaller scale, slices of cooked chips have been examined to determine lignin content variability. Unfortunately, few data are available on lignin content variability within and between individual fibers. Available data do suggest, however, that a high level of interfiber delignification diversity should be present. This study has measured lignin content variability between individual kraft softwood pulp fibers using density gradient column distributions. Large variations in fiber-to-fiber lignin content were observed in pulps from 10.0-mm-thick chips relative to pulps from 2.5-mm-thick chips. Mill-produced pulps were intermediate in delignification diversity. Lo-Solids® pulps were found to show a much higher interfiber uniformity than conventionally produced pulps.*

Traditionally, bleachable grade softwood pulp has been delignified to a kappa number of approximately 30 to optimize strength, yield, and production capacity. Recently, the trend has been to pulp to lower kappa numbers in an effort to minimize bleach plant effluent. Losses in pulp production, yield, and quality can occur from pulping to low residual lignin contents and have been shown to relate to pulping nonuniformities. Thus, oxygen prebleaching from 20+ kappa number is currently favored over extended pulping.¹ A measure of the variation in single-fiber lignin content may lead to a better understanding of pulping limitations, thus minimizing losses.

Nonuniform pulp is a result of variations within and between digesters.^{2,3} Variations between digesters can result from fluctuations in chemical charge, H-factor calculation, furnish swings, etc. Within digester variation may result from oversized chips, inhomogeneous or insufficient liquor flux, and unequal temperature and/or chemical distribution inside and outside of the chips.^{4,5} Improvements in uniformity generally imply that there are less overcooked and undercooked chips,² and result in

pulps of higher strength and yield, along with reductions in bleaching chemical demand.^{2,4,6,7} Unbleached laboratory pulps are often considered the realistic quality potential of a given fiber source.^{5,8,9}

BATCH DIGESTER STUDIES

Blume¹⁰ studied process variations as early as the 1950s, using a "hanging-basket" within batch digesters. Subsequently, MacLeod *et al.*^{5,9,11} measured pulp quality in batch digesters using similar forms of this technique. It was discovered that pulp inside a batch digester is capable of having strength values near those of laboratory pulps. After blowing, pulp strength can be 20-30% lower¹¹. Similarly, Tikka *et al.*,¹² Anderson and Rea,¹³ and Gullichsen *et al.*⁴ each used three-basket arrays to measure variations in pulp strength and lignin content within batch digesters.

CONTINUOUS DIGESTER STUDIES

The only studies published on pulp variation within continuous digesters were performed over 30 years ago by Knutsson¹⁴ and Annergren.¹⁵ This work used a through-the-wall sampling technique developed by Jansson.¹⁶ Similar to the later findings of MacLeod,¹¹ high-quality pulp was present prior to discharge from the digester.

Lignin content variation has been observed using on-line kappa number analyzers.^{17,18} A constant fluctuation of at least ± 5 -10% has been observed,¹⁸ but did not coincide with standard kappa number profiles. This suggests that the mixed stock may be masking a significant lignin content variation existing between fibers.⁵

THE DENSITY GRADIENT COLUMN

A key element in the analysis of the lignin content variation between fibers has made use of the density gradient column concept.^{19,20} Essentially, the density gradient column is a mixture of completely miscible solvents whose composition and density vary with column height. Density gradient columns are very sensitive methods to measure density distributions, and have the ability to differentiate density differences down to about 10^{-7} g/mL.¹⁹

All density separation techniques for wood assume that the individual densities of wood fiber components are additive, where lignin, holocellulose, and α -cellulose have densities of 1.335 g/mL, 1.521 g/mL, and 1.528 g/mL, respectively.²¹ Therefore, the density of an unbleached fiber is assumed to be inversely related to its content of non-cellulosic material, especially lignin.

Paulson was the first to measure interfiber diversity within pulps using a density gradient column.²² Tichy used this method to define, mix, and measure the quality of nonuniform pulps⁶ and Horng, to define and explain nonuniformities in mill produced pulps.⁸ Wandelt and Mroz have used a similar pulp

* Lo-Solids is a registered trademark and Lo-Level is a trademark of Ahlstrom Machinery Inc.

density method to estimate yield from NSSC pulps.²³ Other investigations of pulp uniformity have included UV microspectroscopy²⁴ and fluorescence microphotometry.²⁵ Pulp quality inferences have been made by mixing pulps with assumed levels of uniformity,^{26,27} and a patent was granted for UV fluorescence applications.²⁸

EXPERIMENTAL METHODS

A range of laboratory kraft pulps were produced from 2.5-mm-thick chips and 10.0-mm-thick chips to assess the dependence of interfiber uniformity on diffusion limitations. Pulp lignin contents varied from a maximum of approximately 60 kappa number to the minimum achievable kappa number for each chip furnish. All pulp samples were produced from 500 g o.d. chips. Chips were presteamed for 2 hours under atmospheric pressure. A water aspirator vacuum was used to assist in chip impregnation when necessary. Cooks were carried out at a constant EA of 40 gpl with a L:W ratio of 6:1. This corresponds to a 24% charge on wood. A sulfidity of 30%, based on AA, was used for all cooks. The pulping temperature and time were varied to achieve desired H-factors.

Mill-produced pulps were obtained from Ahlstrom Machinery Inc. and originated from the following series of continuous digester modifications: 1) conventional feed and conventional cooking, 2) conventional feed and Lo-solids[®] cooking,^{29,30} and 3) Lo-level[™] feed and Lo-solids[®] cooking.³¹

The density gradient column is formed from a mixture of chloroform and tetrachloroethylene. A 1-2 mg o.d. pulp sample is freeze-dried and vacuum impregnated with 40 mL of carefully dried chloroform. The fibers are dispersed, and transferred to the bottom of a dried density gradient column. Gradient formation occurs underneath the sample while floating the fibers and chloroform under a pad of dry N₂. Columns were developed for two days before imaging. Imaging and data acquisition from the column was achieved through cross-polarization techniques and digital image averaging. Optimas[™] image analysis software was used to determine relative fiber density distributions.

RESULTS

Pulping Results

Table 1 summarizes the laboratory produced kraft pulps used in this study. A liquor to wood ratio of 6:1 was chosen to enhance mass transfer of cooking chemicals into the hand-cut chips. All pulps were subject to chlorite holopulping and these samples serve as lignin-free pulp standards for the sample set.³²

The pulps in Table 1 came from a set of 15 pulps whose characteristics are further described in Figures 1 and 2. Figure 1 shows that a larger amount of lignin removal occurred at a given H-factor with 2.5-mm-thick chips. The alkali had a

shorter diffusion distance to the reaction site, and dissolved lignin was required to leach through a shorter diffusion distance from inside the chip into the cooking medium. Moreover, it has been proposed that a wider alkali concentration gradient exists between the outer chip boundary and center for 10.0-mm chips than for 2.5-mm-thick chips.^{33,34}

Table 1. Description of laboratory pulps.

| Parameter | 1 | 2 | 3 | 4 | 7 | 9 |
|-----------------------------|-------|-------|-------|-------|-------|-------|
| chip thickness, mm | 10.0 | 2.5 | 10.0 | 2.5 | 10.0 | 2.5 |
| temperature, C | 150 | 150 | 170 | 165 | 160 | 154 |
| H-factor | 548 | 548 | 2803 | 1892 | 1274 | 786 |
| kappa number | 63.7 | 54.6 | 23.4 | 15.9 | 33.4 | 33.9 |
| viscosity, cP | 46.07 | 44.97 | 16.88 | 17.19 | 37.58 | |
| yield, unscreened | 0.520 | 0.442 | 0.423 | 0.394 | 0.482 | 0.455 |
| yield, screened | 0.515 | 0.442 | 0.413 | 0.394 | 0.474 | 0.455 |
| time to temperature, min. | 60 | 60 | 60 | 60 | 60 | 60 |
| time at temperature, min. | 240 | 240 | 240 | 240 | 240 | 240 |
| L:W=6:1, EA=24% on wood | | | | | | |
| 30% sulfidity (based on AA) | | | | | | |

Table 2. Description of mill pulps.

| Description | Kappa Number | Viscosity, cP |
|-------------------|--------------|---------------|
| conventional cook | 31.4 | 34.7 |
| Lo-Level feed | 24.6 | 27.3 |
| Lo-Solids cook | | |
| conventional feed | 24.1 | 22.2 |
| Lo-Solids cook | | |

The pulps prepared from 2.5-mm-thick chips were of substantially higher viscosity than pulps from 10.0-mm-thick chips (Figure 2). This viscosity difference was assumed to result from overcooking of the chip perimeter.^{35,36,37,38} Early research by Harlter *et al.* did not show viscosity differences in 3-7 mm thickness chips.^{36,37} More recent work by Akhtaruzzaman *et al.*³⁵ and Gullichsen³⁸ did identify that viscosity decreases markedly at a given kappa number with an increase in chip thickness.

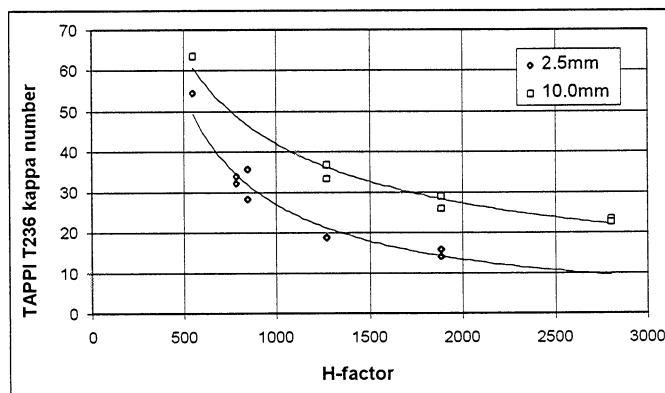


Figure 1. TAPPI T236 kappa number vs. H-factor for laboratory pulps.

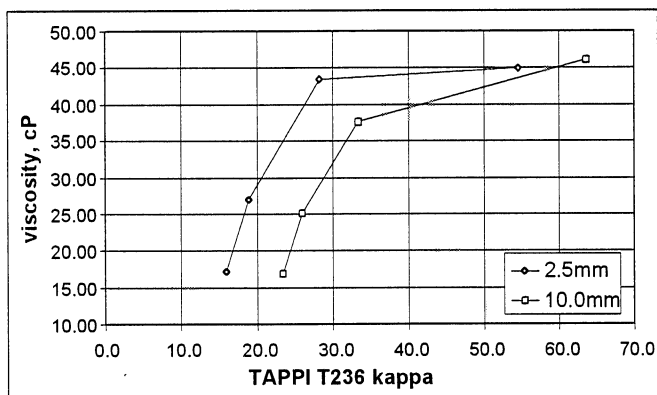
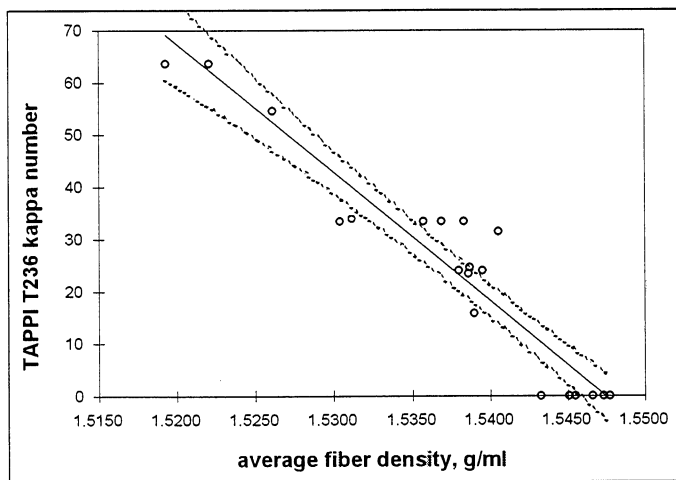


Figure 2. Viscosity vs. T236 kappa number for laboratory pulps prepared from thin and thick chips.

The results in Figure 2 compare differences in viscosity and kappa number obtained from extreme differences in chip thicknesses. Density gradient column distributions were successful in providing further insight into the pulping limitations that create these differences.

Analyses of Kappa Number and Lignin Density

Figure 3 relates average fiber density to kappa number. The kappa number vs. average fiber density relationship provides a method of converting individual fiber density values to an estimated density gradient column kappa number equivalent (DGC kappa number). This provides the basis for all analyses of delignification diversity.



| slope | intercept | R ² | std. error in kappa number |
|-----------|-----------|----------------|----------------------------|
| -2.46E+03 | 3.80E+03 | 0.907 | 6.5 |

Figure 3. TAPPI T236 kappa number vs. average fiber density.

Table 3 provides the densities of lignin and holocellulose fiber components, as determined by the density gradient column analyses. Holocellulose densities were independent of extent of cook and process condition within the pooled random error.

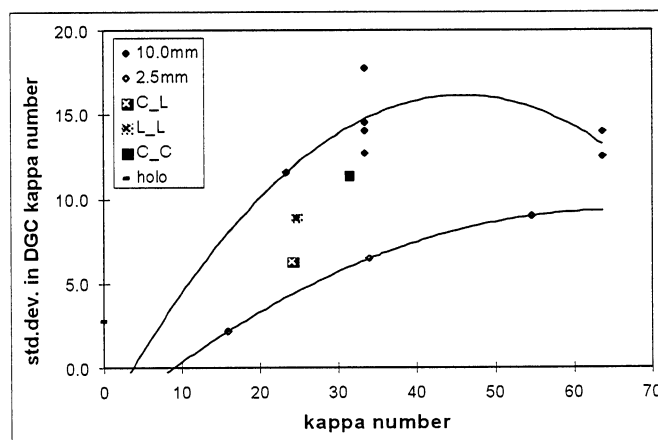
Table 3. Density of holocellulose and lignin.

| component | density (g/mL) | std. deviation (g/mL) |
|---------------|-------------------|--------------------------|
| holocellulose | 1.5458 | 0.0011 |
| lignin | 1.2719 | 0.0090 |

These values are 2% higher for holocellulose and 5% lower for lignin than the values reported by Stamm.²¹ This is thought to be due primarily to the extra effort made to dry solvents and fibers in this study.

Measurement of Delignification Diversity

Figure 4 illustrates delignification diversity in terms of the overall standard deviation in DGC kappa number for pulps prepared from 2.5-mm-thick chips, 10.0-mm-thick chips, and the mill-produced pulps. The pulps produced from 10-mm-thick chips show about twice the standard deviation in DGC kappa number of the pulps produced from 2.5-mm-thick chips. Of the three mill pulps, the conventional continuous digester pulp at 33 kappa number has almost as large a standard deviation as the laboratory pulps produced from 10-mm-thick chips. The level of uniformity of the pulp produced with the digester operating as a Lo-solids vessel is nearly as good as the laboratory pulps prepared from 2.5-mm-thick chips.



C_L refers to conventional feed and Lo-solids® cooking.
L_L refers to Lo-level™ feed and Lo-solids® cooking.
C_C refers to conventional feed and conventional cooking.
2.5mm and 10.0mm refer to chip thickness of laboratory kraft pulps.
Holo refers to the standard deviation in average fiber density for a wide array of kraft holopulps.

Figure 4. Overall standard deviation in DGC kappa number vs. TAPPI T236 kappa number.

Figure 5 shows the individual fiber DGC kappa number distribution of a 33 kappa number laboratory pulp prepared from 10-mm-thick wood chips, and Figure 6 for a 34 kappa number pulp produced from 2.5-mm-thick wood chips. Clearly, the lignin or kappa number distribution in the thin chip sample is much narrower than that in the thick chip sample.

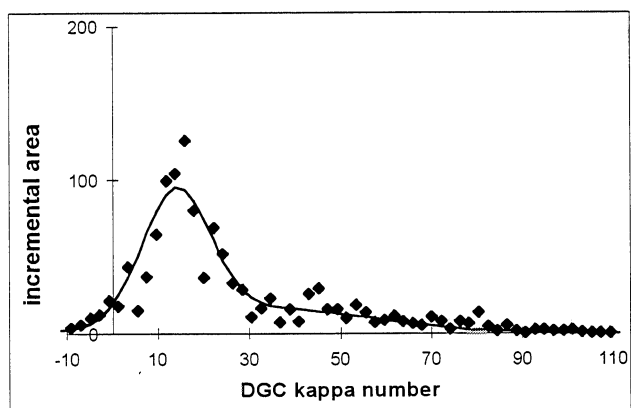


Figure 5. DGC kappa number distribution of 33.4 kappa number laboratory pulp from 10-mm-thick chips.

The majority of the fibers in the thick chip distribution are centered around a 14 DGC kappa feature with a normal distribution. In addition, there is a large tail extending to values in excess of a 70 DGC kappa number. Although the average DGC kappa for the sample is about 30, many of the fibers are at very low residual lignin contents suggesting an overcooked condition. The equivalent laboratory cook of 2.5-mm-thick chips at 160° C and 1274 H-factor produced a T-236 kappa value of 18.9. This is similar to the normal part of the distribution shown in Figure 5. The thin chip sample (Figure 6) has a much smaller tail to high kappa numbers and the normal part of the distribution is centered very close to the chemical kappa value.

The individual fiber DGC kappa number distribution for 3 samples at different T-236 kappa numbers is shown in Figure 7 for samples prepared from 2.5-mm-thick chips, and Figure 8 for samples from 10-mm-thick chips. For these figures, a normal distribution is fitted to the "normal" part of the data. The pulp samples prepared from thin chips show a regular progression of the distribution to lower DGC kappa. The

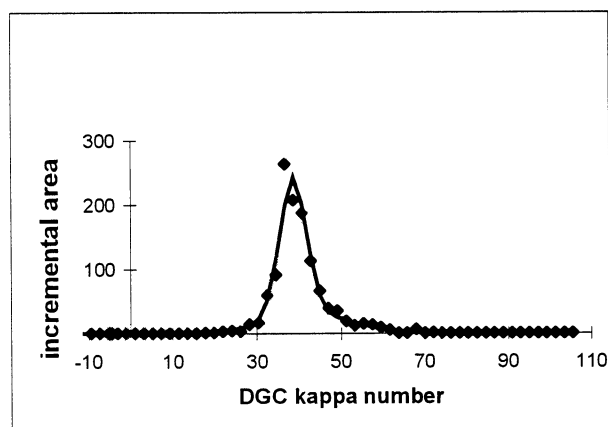


Figure 6. DGC kappa number distribution of fibers from a 33.9 kappa number laboratory cook of 2.5-mm-thick chips.

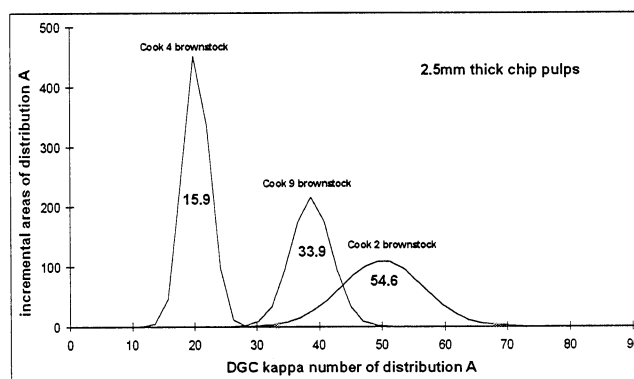


Figure 7. The "normal" portion of the DGC kappa number distribution for a variety of cooks performed on 2.5-mm-thick chips.

distributions become sharper (decrease in standard deviations) as cooking progresses. This is not observed in the pulps produced from thick chips. The distributions are quite broad, even at low kappa numbers. In addition, the mean values are very similar for the 33.4 kappa and 23.4 kappa value pulps. This is because the pulping conditions for these samples are suitable for producing a ~ 20 kappa pulp under conditions with uniform distribution of chemicals. The normal portion of the distribution appears to arise from the perimeter of the chips where access to cooking chemicals is not impeded.

The lignin distribution for two of the three continuous digester samples is shown in Figures 9 and 10. The 33 kappa number sample from conventional digester operation shows a relatively broad distribution with a pronounced tail. Referring to Figure 4,

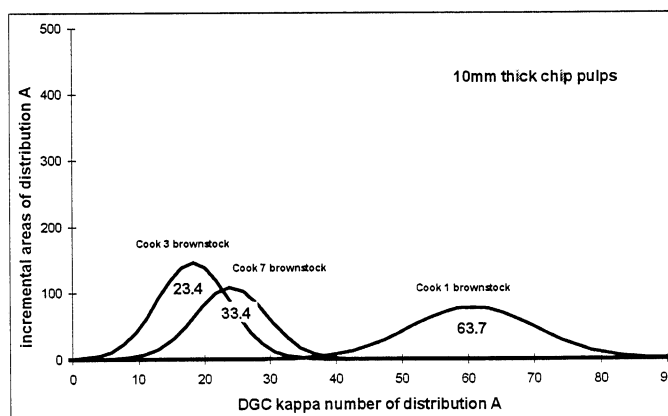


Figure 8. "Normal" portion of the DGC kappa number distribution for pulps prepared from 10-mm-thick chips.

the standard deviation is between the response curves determined for the lab pulps produced from the 2.5-mm and 10-mm-thick chips, but the closest of the mill pulps to the 10-mm-thick chip curve. The pulp sample produced with the digester operating as a Lo-solids digester shows a significant narrowing of the distribution (Figure 10) and shows a standard deviation

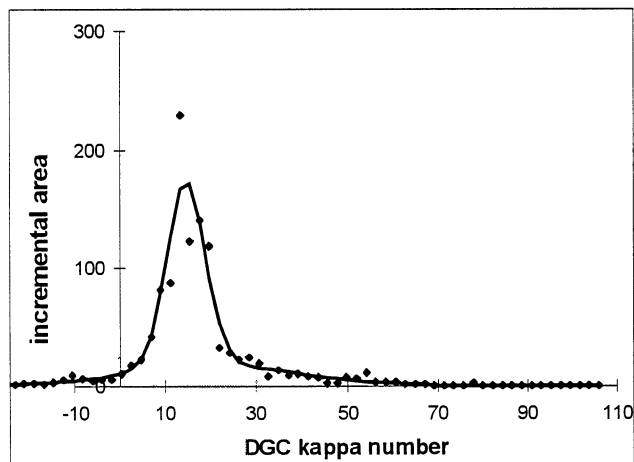


Figure 9. The single fiber DGC kappa number distribution for a pulp sample from a conventional continuous digester. Sample is 33.4 kappa number.

(Figure 4) close to the response curve for the laboratory pulps produced from the 2.5-mm-thick chips. The pulp sample obtained with the digester operating in a Lo-solids, Lo-level feed mode gave a distribution between these two extremes.

CONCLUSIONS

The density gradient column technique has proven useful for determining lignin variation on an individual fiber basis in kraft pulps. Pulp samples produced from 10-mm-thick chips show significantly larger standard deviations in density gradient column kappa values and a pronounced tail of poorly delignified fibers. Pulp samples produced from 2.5-mm-thick wood chips show a much sharper distribution and a much smaller high-lignin-content tail. Three continuous digester samples have also been tested. A conventional cook sample at kappa number 33 is much like the thick chip laboratory samples. It gives a broad distribution with a pronounced tail. A sample provided from a digester operating in Lo-solids mode gave a much more uniform pulp with a minimal high kappa value tail. The third sample produced in a digester operating in Lo-solids Lo-level feed mode gave a DGC kappa distribution between the other two.

ACKNOWLEDGEMENTS

Portions of this research were used by B.S. Boyer as partial fulfillment of the Ph.D. degree at the Institute of Paper Science and Technology. Dr. Boyer is currently pursuing a degree in law at the John Marshall Law School, Chicago, IL. We would like to thank the Institute of Paper Science and Technology and its member companies for supporting this research. Special thanks go to research committee members Dr. Earl Malcolm, Dr. Thomas McDonough, Dr. Hiroki Nanko, and Dr. Jian Li. Additional thanks go to Dr. Bruno Marcoccia of Ahlstrom Machinery Inc. for providing the mill samples.

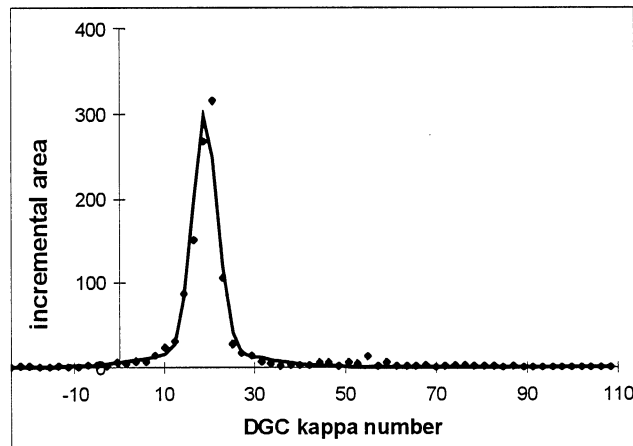


Figure 10. DGC kappa number distribution from a 24.1 kappa number pulp produced in a Lo-solids® continuous digester.

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